

sample, remove the probe from the stack and conduct a leak-check according to the procedures outlined in Section 8.2 of Method 6 (mandatory). After each 3-hour test run (or after three 1-hour samples), conduct one system performance check (see Section 8.5). After this system performance check and before the next test run, it is recommended that the probe be rinsed and brushed and the filter replaced.

NOTE: In Method 15, a test run is composed of 16 individual analyses (injects) performed over a period of not less than 3 hours or more than 6 hours. For Method 15A to be consistent with Method 15, the following may be used to obtain a test run: (1) Collect three 60-minute samples or (2) collect one 3-hour sample. (Three test runs constitute a test.)

8.4 Sample Recovery. Recover the hydrogen peroxide-containing impingers as detailed in Method 6, Section 8.4.

8.5 System Performance Check.

8.5.1 A system performance check is done (1) to validate the sampling train components and procedure (before testing, optional) and (2) to validate a test run (after a run, mandatory). Perform a check in the field before testing consisting of at least two samples (optional), and perform an additional check after each 3-hour run or after three 1-hour samples (mandatory).

8.5.2 The checks involve sampling a known concentration of COS and comparing the analyzed concentration with the known

concentration. Mix the recovery gas with N₂ as shown in Figure 15A-4 if dilution is required. Adjust the flow rates to generate a COS concentration in the range of the stack gas or within 20 percent of the applicable standard at a total flow rate of at least 2.5 L/min (5.3 ft³/hr). Use Equation 15A-4 (see Section 12.5) to calculate the concentration of recovery gas generated. Calibrate the flow rate from both sources with a soap bubble flow tube so that the diluted concentration of COS can be accurately calculated. Collect 30-minute samples, and analyze in the same manner as the emission samples. Collect the samples through the probe of the sampling train using a manifold or some other suitable device that will ensure extraction of a representative sample.

8.5.3 The recovery check must be performed in the field before replacing the particulate filter and before cleaning the probe. A sample recovery of 100 ± 20 percent must be obtained for the data to be valid and should be reported with the emission data, but should not be used to correct the data. However, if the performance check results do not affect the compliance or noncompliance status of the affected facility, the Administrator may decide to accept the results of the compliance test. Use Equation 15A-5 (see Section 12.6) to calculate the recovery efficiency.

9.0 Quality Control

Section	Quality control measure	Effect
8.5	System performance check	Ensures validity of sampling train components and analytical procedure.
8.2, 10.0	Sampling equipment leak-check and calibration.	Ensures accurate measurement of stack gas flow rate, sample volume
10.0	Barium standard solution standardization	Ensures precision of normality determination.
11.1	Replicate titrations	Ensures precision of titration determinations.
11.2	Audit sample analysis	Evaluates analyst's technique and standards preparation.

10.0 Calibration and Standardization

10.1 Metering System, Temperature Sensors, Barometer, and Barium Perchlorate Solution. Same as Method 6, Sections 10.1, 10.2, 10.4, and 10.5, respectively.

10.2 Rate Meter. Calibrate with a bubble flow tube.

11.0 Analytical Procedure

11.1 Sample Loss Check and Sample Analysis. Same as Method 6, Sections 11.1 and 11.2.

11.2 Audit Sample Analysis. Same as Method 6, Section 11.3.

12.0 Data Analysis and Calculations

In the calculations, retain at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculations.

12.1 Nomenclature.

C_{COS} = Concentration of COS recovery gas, ppm.

C_{RG(act)} = Actual concentration of recovery check gas (after dilution), ppm.

C_{RG(m)} = Measured concentration of recovery check gas generated, ppm.

C_{RS} = Concentration of reduced sulfur compounds as SO₂, dry basis, corrected to standard conditions, ppm.

N = Normality of barium perchlorate titrant, milliequivalents/ml.

P_{bar} = Barometric pressure at exit orifice of the dry gas meter, mm Hg.

P_{std} = Standard absolute pressure, 760 mm Hg.

Q_{COS} = Flow rate of COS recovery gas, liters/min.

Q_N = Flow rate of diluent N₂, liters/min.

R = Recovery efficiency for the system performance check, percent.

T_m = Average dry gas meter absolute temperature, °K.

T_{std} = Standard absolute temperature, 293 °K.

V_a = Volume of sample aliquot titrated, ml.

V_{ms} = Dry gas volume as measured by the sample train dry gas meter, liters.

V_{mc} = Dry gas volume as measured by the combustion air dry gas meter, liters.

$V_{ms(std)}$ = Dry gas volume measured by the sample train dry gas meter, corrected to standard conditions, liters.

$V_{mc(std)}$ = Dry gas volume measured by the combustion air dry gas meter, corrected to standard conditions, liters.

V_{soln} = Total volume of solution in which the sulfur dioxide sample is contained, 100 ml.

V_t = Volume of barium perchlorate titrant used for the sample (average of replicate titrations), ml.

V_{tb} = Volume of barium perchlorate titrant used for the blank, ml.

Y = Calibration factor for sampling train dry gas meter.

Y_c = Calibration factor for combustion air dry gas meter.

32.03 = Equivalent weight of sulfur dioxide, mg/meq.

$$12025 \frac{\mu\text{l}}{\text{meq}} = \frac{(32.03 \text{ mg})}{\text{meq}} \frac{(24.051)}{\text{mole}} \frac{(\text{mole})(1\text{g})(10^3 \text{ ml})(10^3 \mu\text{l})}{(64.06\text{g})(10^3 \text{ mg})(1)(\text{ml})}$$

12.2 Dry Sample Gas Volume, Corrected to Standard Conditions.

$$\begin{aligned} V_{m(std)} &= V_{ms} Y \frac{T_{std} P_{bar}}{T_m P_{std}} & \text{Eq. 15A-1} \\ &= K_1 Y \frac{V_m P_{bar}}{T_m} \end{aligned}$$

Where:

K_1 = 0.3855 °K/mm Hg for metric units,

= 17.65 °R/in. Hg for English units.

12.3 Combustion Air Gas Volume, corrected to Standard Conditions.

$$V_{mc(std)} = k_1 Y_c \frac{(V_{mc})(P_{bar})}{T_m} \quad \text{Eq. 15A-2}$$

NOTE: Correct P_{bar} for the average pressure of the manometer during the sampling period.

12.4 Concentration of reduced sulfur compounds as ppm SO_2 .

$$C_{RS} = K_2 N \frac{(V_t - V_{tb})(V_{soln}/V_a)}{V_{ms(std)} - V_{mc(std)}} \quad \text{Eq. 15A-3}$$

Where:

$$\begin{aligned} K_2 &= 32.03 \frac{\text{mg}}{\text{meq}} \frac{24.05 \text{ l}}{\text{mole}} \frac{1 \text{ mole}}{64.06 \text{ g}} \frac{1 \text{ g}}{10^3 \text{ mg}} \frac{10^3 \text{ ml}}{1} \frac{10^3 \mu\text{l}}{\text{ml}} \\ &= \frac{12025 \text{ ml}}{\text{meq}} \end{aligned}$$